MEMS Near-Infrared Reflectance Absorption Spectrometer for Detection of Composition in Soil

Yunbo Shi, Xiaoyu Yu, Debin Xiu, Qiaohua Feng, Wenjie Zhao, Changbin Tao

Abstract

A near infrared absorption spectrometer using MEMS near infrared light source has been assembled. This system was evaluated and found attractive for detection of the composition in soils using a MEMS near infrared light source in the reflectance absorption spectrometer. Analysis and experiments show that, with the traditional laboratory chemical testing technology, the system has advantages of small size, low cost, no pollution, portable, nondestructive, etc, greatly improves the working efficiency of the agricultural science and technology personnel, reduced the working intensity, that do not have the laboratory condition of rural also can be easily for testing.

Keywords: MEMS Light Source, Near-Infrared Reflectance Spectrometer, Soil Composition Detection

1. Introduction

Soil composition like the trace elements N, P, K, organic matter and moisture can directly affect the growth of crops. The chemical analysis method has always been to detect the soil composition content in a traditional situation. Detection of the composition in the soil requires an innovative technique which has low-cost, real-time advantages. The use of MEMS light source in conjunction with reflectance spectroscopy for characterizing soil composition detection provides one such technique.

The purpose of this work is to report on the assembly and evaluation of a laboratory near-infrared (NIR) reflectance spectrometer for low-cost, real-time detection and characterization of trace elements and organics in soils. This system includes the MEMS light source, the grating light path, measurement amplifier circuit, data acquisition and diver modules, chemical analysis software, all of which are commercially available components. Reflectance spectroscopy is well suited for such measurements, because it can detect and identify both the trace elements and organics, and does not require sample handing or preparation. This eliminates the need for taking the soil sample to the chemical laboratory for analyzing. The NIR spectral region used, between 0.8 and 2.5μm, is convenient because of using the conventional light sources and sensitive detectors. This paper reports the results of a preliminary investigation involving of this reflectance spectrophotometer using a MEMS light source. This investigation clearly demonstrates the possibility of using a MEMS light source in the NIR reflectance spectrometer.

The quantitative analysis of near infrared spectral technology is the measuring technique using of near infrared light to illuminate the sample, and then analyses the reflected light carrying the material information, thus quickly assesses just one or several component content. Reflectance spectroscopy is
to stay test sample and light detector in the same side, detector detects the light that is returned from the sample in a variety of ways. Diffuse light is the phenomenon that the no rules reflection light illuminated to rough surface. When a light illuminated to a certain thickness of granular material sample layer, and some of those was absorbed, and the other part was reflected at last with the sample all levels by internal reflection, absorption, refraction. Diffuse reflected light carrying the sample information and the characteristics of internal sample [1], [2]. Strictly speaking, the reflection of near infrared spectral method should be diffuse near infrared spectral method. In the near infrared spectrum analysis, the measurement method is general used to detect the solid sample.

Diffuse rate R and sample components concentration are not linear relationship, there are two kinds of the commonly used diffuse spectrum function have relationship with components concentration, namely launch absorbency and Kubelka-Munk diffuse absorbency:

\[
A = \log \left[ \frac{1}{R_\infty} \right] = -\log \left[ 1 + \frac{K}{S} \sqrt{\frac{K}{S}} + 2 \left( \frac{K}{S} \right) \right]
\]

A: Diffuse reflection absorbance of the diffuse body

\( R_\infty \): Diffuse rate of the diffuse body

K: Absorption coefficient of the diffuse body

S: Scattering coefficient of the diffuse body

When the K/S in a certain range, A and K/S can use linear relationship instead of curve relationship.

\[ A = a + b(K/S) \]

The K/S function of diffuse spectrum:

\[ F(R_\infty) = K/S = bc \]

Constant b, and sample molar absorption coefficient and optical path and other relevant; c for sample component concentration. Namely K/S function and samples concentration are direct ratio [3].

2. Experimental details

As a practical portable optical instrument, determine the workings of the instrument as follows. A specific wavelength incident light from the MEMS light source illuminates the collimating object lens, the collimating light changed into angle monochrome collimated light through the plane blazed grating diffraction, then through a focusing object lens reflects the light to a focus at the sample surface, in order to obtain the spectral wavelength lines. The incident light arrived the sample soil surface, a part was absorbed by the soil, and the other part occured diffuse reflection. If the wavelength of the monochromatic light was the sensitive wavelength of the soil organic matter, the light that was absorbed was directly proportional to the content of the soil organic matter, in other words the diffuse reflection light intensity was inversely proportional to the content of soil organic matter and trace elements. Therefore, through the measurement of diffuse reflection light intensity of the soil can indirectly measure the content of soil organic matter and trace elements [4], [5].
Figure 1. A schematic representation of the NIR reflectance absorption spectrometer

Fig.1 shows a schematic representation of the layout used for the prototype system. The optical system consists of the aforementioned MEMS light source, collimating object lens, blazed grating, focusing object lens and near infrared detector. And the signal processing system consists of the amplifying circuit, phase sensitive detection and MCU, etc.

System adopts grating spectral institutions, the plane blazed grating for spectrophotometry element. Blazed grating requires that the incident light is parallel light, the parallel composite light diffracted into different angle monochrome parallel light. So there is a collimator object lens between the entrance slit and grating to change the incident light into parallel light, the parallel light is then focused on the image surface after diffracted by a focusing object lens, to obtain the order wavelength of the spectral lines [6], [7], [8].

The entrance slit should be located the focal plane of the collimator objective, and the exit slit was the imaging of the entrance slit, should focus on the focal plane of the focusing objective in place. Selected two slit width of 0.1 mm, high of 2.5 mm, to ensure that the incident light from entrance slit all shining on the collimator objective and diffraction of all also as monochromatic light after the focus on the focusing objective, so the two spherical reflector size option for 30 mm × 30 mm, the focal length \( f = 50 \text{ mm} \).

Reflectance was measured in a diffuse mode in which the normal to the plane of the sample was offset 20° relative to the plane formed by the mirrors and sample. Furthermore, all spectra were measured using the intensity spectrum of the typical Northeast black soil in China in diffuse mode. Sampling depth was the range of 0–20cm after removing the surface soil, the weight of each soil sample was between 1.4–3.2 kg, in order to guarantee the representative and sampling uniformity, in each vertex and central point of the soil sample collection area two diagonals collected five soil samples, fully blending as soil samples.
In order to reduce human errors, evenly spreading out the soil samples collected then let it dry naturally. The dried soil samples which was grinded by a ball grinding mill that was filtered with 1 mm sieve. The soil samples were encapsulated in hermetically sealed containers.

Because for the measured signal and the reference signal through different circuit to enter the correlator, which may produce a certain phase difference, in order to eliminate the phase difference, make the lock-in amplifier output reached the maximum, design phase shifting circuit.

\[ H_s(j\omega) = \frac{1 - j\omega R_s C}{1 + j\omega R_s C} \]  

(2)

Which can be used to obtain the phase frequency response:

\[ \phi(\omega) = -2 \tan^{-1}(\omega R_s C) \]  

(3)

The biggest phase shift absolute value is less than 180, choose \( R_s = 5k\Omega, C = 1\mu F \), In \( \omega_0 = 2\pi f_0 \) cases,

\[ |\phi_{\text{max}}(\omega)| = 136^\circ \]  

(4)

The MEMS light source chosen rectangular cell using dry etching method in the back of the SOI chip for smaller area. In the oxidation silicon layer etched the electrode windows and sputtered the aluminum electrodes. In order to reduce energy consumption of the MEMS light source, the monocrystalline silicon layer at the top of the SOI chip was designed as infrared absorption layer of heavily doped, absorbing the radiation from the back of the luminescent film to store energy, achieving the effect for self-heating. [9], [10]The MEMS light source was manufactured by photolithography process, magnetron sputtering, and dry etching. In addition, the wavelength of the MEMS light source between 0.5 \( \mu m \) and 4 \( \mu m \), the modulation frequency can be as high as 40 Hz at 50 % modulation depth.

Because of the near infrared spectral area is a bit wide, sample spectrum was overlap, it is difficult to detect the absorption characteristics of the sample components, so classic quantitative analysis is not suitable for complex samples. Now commonly uses the metrology correction method to overcome the
shortcomings of the classic method. Such as Least-squares regression method can effectively overcome
the information overlap caused by the multiple correlation, and improve the accuracy of the models.

Due to the fact that the measurement of spectral data includes not only the test sample composition
information, and also includes high frequency noise and low frequency noise and drift caused by the
instrument background or drift, so study adopts digital Fourier filter pretreatment method. First data
will be Fast Fourier Transform (FFT), in frequency space and Gaussian window function, and then the
Fast Fourier Transform (IFFT), to get the spectral data from the band-pass filter. The mean and
standard deviation of Gaussian function respectively determine the center frequency and bandwidth of
the band-pass filter, in the study the numerical optimization method to determine the two parameters
for optimal filtering effect. This method can effectively increase the spectral signal-to-noise ratio [11],
[12].

In addition, genetic algorithm is applied to the optimization of wavelength variables. Because
eliminate the variable of the do not related or non-linear variable, the prediction capability of the model
is improved, and applies to pure PLS more difficult to correction associated system. At the same time
as the number of modeling variables decreases, can greatly reduce measuring time and system
resources. Using the Visual C++ 6.0 to write the program of the genetic algorithm variable
optimization in the study. PLS correction algorithm program and digital Fourier filter program is used
MATLAB5.3 to write. The near infrared spectral analysis technology, although in establishing a
correction models need to spend some time, but when the model was settled, the test is very fast, a
spectrum measurement of the sample and component content prediction can be in a few seconds, so
especially suitable for online application.

3. Experimental results and discussion

![Figure 3. The prediction model of the soil total nitrogen content](image)

Fig. 2 shows the prediction model using partial least squares prediction model of the soil total
nitrogen content, the determination coefficient R2 reached 95.44%, root mean square error of cross-
validation (RMSECV) is 0.0141, root mean square error of prediction (RMSEP) is 0.0168, relative
percent deviation (RPD)is 4.67. RPD > 3 shows the stability of the model is good.
Figure 4. Continuous 11 times measurement reflectance spectrum and its MSE

The instrument stability of the output signal is to point to in the working conditions of the instrument stable degree, used to evaluate the mean-square error of the spectrum of scanning the sample many times on the specified range of wavelengths.

Fig. 3 for boot after stability, measuring the background, a white board, and continuous measured the same sample 11 times obtain reflectance spectrum Y, its relative mean-square error of spectrum mean-square error STD (Y), relative spectrum mean (Y), restd (Y) = STD (Y) / mean (Y), all the maximum mean-square error is 0.00777, the maximum relative mean-square error 0.01704, the instrument stability error \( \leq 1.704\% \). If eliminating the last most unstable four data, its biggest mean-square error value is 0.00394, the maximum relative mean-square error is 0.00839, the instrument stability error \( \leq 0.839\% \).

<table>
<thead>
<tr>
<th>Order number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tr>
<td>maximum</td>
<td>19478</td>
<td>19479</td>
<td>20381</td>
<td>19660</td>
<td>17602</td>
<td>18112</td>
<td>16509</td>
<td>18335</td>
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<tr>
<td>Corresponding wavelength ( \lambda )</td>
<td>1896</td>
<td>1896</td>
<td>1896</td>
<td>1900</td>
<td>1896</td>
<td>1900</td>
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<td>1900</td>
</tr>
<tr>
<td>Half peak wavelength( \lambda_{1} )</td>
<td>1890.7</td>
<td>1890.7</td>
<td>1890.0</td>
<td>1893.5</td>
<td>1890.1</td>
<td>1893.7</td>
<td>1890.3</td>
<td>1893.9</td>
</tr>
<tr>
<td>Half peak wavelength( \lambda_{2} )</td>
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<td>1902.7</td>
<td>1902.0</td>
<td>1905.5</td>
<td>1902.1</td>
<td>1905.7</td>
<td>1902.3</td>
<td>1905.9</td>
</tr>
<tr>
<td>Half peak width ( \lambda_{1})–( \lambda_{2} )</td>
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<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
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<td>12.0</td>
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<tr>
<td>( \lambda_{1})–( \lambda_{2} )</td>
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<td>-2.4</td>
<td>-2.4</td>
<td>1.6</td>
<td>-2.4</td>
<td>1.6</td>
<td>-2.4</td>
<td>1.2</td>
</tr>
</tbody>
</table>

The wavelength of the spectral instrument accuracy is referred to the difference between the instrumental determination standard material a spectral peak wavelength and the peak wavelength calibration of the spectrum. Wavelength reproducibility is referred to scan repeatedly the same sample, the difference between the spectral peaks.

Table 1 shows the continuous measurement of a white board output signal after 20 minutes when the instrument was stable, using a wavelength of 632.8 nm laser as the light source. According to the table 1:

The wavelength accuracy of the instrument \( \leq \max (|\lambda - \lambda|) = 2.4nm \)

The wavelength repeatability of the instrument \( \leq \max (\lambda) - \min (\lambda) = 1900 - 1896 = 4nm \)

4. Conclusion

The study clearly demonstrates the feasibility of detecting the trace elements N, P, K, and organic matters in soils with the reflectance absorption spectrometry using the MEMS near infrared light
source. Improvements to MEMS light source with greater intensity and stability, detector with greater sensitivity and design refinements for achieving better reflectance efficiency. Furthermore, application of reflectance spectroscopy to actual field use would require consideration of additional factors such as miniaturization for devices like field testing.

Thanks to the rapid development of near infrared technology, this paper discusses this technology breakthrough of the realization of near infrared spectral technology used in the soil composition content online measurement field. In line with the future agriculture the development trend to rapid, accurate, green, low cost, fill the blank of the near infrared soil composition content portable detection system. The system modeling coefficient of determination of total nitrogen and organic matter is 0.8324 and 0.8833, the standard error is 0.0097 and 0.0099, enough to satisfy the measurement requirements, so this study is of high prospective and practical value.

5. Acknowledgements

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6. References


